

Helium Mass Spectrometer Leak Detection: A Method to Quantify Total Measurement Uncertainty

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ABSTRACT

In applications where leak rates of components or systems are evaluated against a leak rate requirement, the uncertainty of the measured leak rate must be included in the reported result. However, in the helium mass spectrometer leak detection method, the sensitivity, or resolution, of the instrument is often the only component of the total measurement uncertainty noted when reporting results. To address this shortfall, a measurement uncertainty analysis method was developed that includes the leak detector unit's resolution, repeatability, hysteresis, and drift, along with the uncertainty associated with the calibration standard. In a step-wise process, the method identifies the bias and precision components of the calibration standard, the measurement correction factor (K-factor), and the leak detector unit. Together these individual contributions to error are combined and the total measurement uncertainty is determined using the root-sum-square method. It was found that the precision component contributes more to the total uncertainty than the bias component, but the bias component is not insignificant. For helium mass spectrometer leak rate tests where unit sensitivity alone is not enough, a thorough evaluation of the measurement uncertainty such as the one presented herein should be performed and reported along with the leak rate value.

BACKGROUND

In leak detector tests, it is common to specify the sensitivity of the leak detector unit or determine the minimum detectable leakage rate. These terms are often used inconsistently, and can be a source of confusion. The American Vacuum Society standard AVS 2.1 Rev 1973 ¹ provides a procedure for calibrating mass spectrometer leak detectors (MSLD) that uses sensitivity to determine the minimum detectable leak rate. In contrast, ASTM standard E1316-14² defines "minimum detectable leakage rate" and "sensitivity of leak test" synonymously. In addition, basic procedures for calibrating and performing leak detector tests are described in the ASNT Leak Testing Handbook,³ various ASTM standards,⁴⁻⁶ and manufacturers' publications,⁷ among other sources. Often the manufacturer specifications for a leak detector will include drift, however, it is usually applicable to the most sensitive range of an operating mode and not necessarily the range in which the leak test is being conducted. Unlike other measurement devices, a specification for the overall accuracy of leak detectors is not given and procedures for determining total measurement uncertainty for mass spectrometer leak detector tests are not common.

Just as the measurement testing errors are important in pressure change leak tests,⁸ the measurement errors, or measurement uncertainty, in a leak detector test are also important to understand and include in the reported leak rate. Pryor and Walker describe the measurement errors commonly associated with the pressure change leak test,⁸ and in an analogous way, the measurement errors associated with a helium MSLD test are described herein. In both types of leak tests, reporting a leak rate without its associated uncertainty is incomplete. Furthermore, the leak rates cannot be properly evaluated with respect to the governing leak rate requirement in the absence of associated uncertainty.

When a measurement is taken and read directly from a piece of instrumentation, the uncertainty of the measured value is associated solely with the device. For example, a pressure gauge with 1 psig graduated increments has a measurement uncertainty of 0.5 psig. However, for calculated values where the value is a function of multiple variables, $y = f(x_1, x_2, x_3 \dots)$, the uncertainty of each component ($x_1, x_2, x_3 \dots$) contributes to the total measurement

uncertainty which is reported. This summation of individual errors is known as the propagation of errors. Neglecting the error associated with the covariance of terms (e.g., x_1x_2 , x_2x_3 , etc.), the total uncertainty (σ_y) can be calculated as shown in Equation 1 below.⁹

$$\text{Equation 1} \quad \sigma_y^2 = \sum_{i=1}^N \left(\frac{\partial y}{\partial x_i} \right)^2 \sigma_{x_i}^2 \quad \text{and} \quad \sigma_y = \sqrt{\sigma_y^2}$$

A method was developed to determine the measurement uncertainty of helium leak rates using a helium MSLD based on the propagation of errors. The method is specifically for dynamic helium MSLD leak tests³ although portions are applicable to other leak tests performed with a MSLD. The measurement errors associated with the leak test are classified as either bias errors or precision errors. The bias, also referred to as systematic error, can be thought of as the off-set between the measured value and the actual value, while precision terms provide information regarding how well the measurement was taken.^{9,10} The root-sum-square method, or RSS method, is then employed to combine the total bias contribution with the total precision contributions as shown in Equation 2 where σ_T is the total uncertainty, σ_β is the total bias contribution to the uncertainty, and σ_ϕ is the total precision contribution to the uncertainty.

$$\text{Equation 2} \quad \sigma_T = \sqrt{(\sigma_\beta^2 + \sigma_\phi^2)}$$

METHOD DEVELOPMENT

When using a helium MSLD to quantify the leak rate of a component or system, the addition of knowing the reliability of the measurement provides a complete description of the performance with respect to leak rate. The reliability is expressed as the uncertainty, however, uncertainty of MSLD measurements is not a common specification from the manufacturer. To address this deficiency, a five step method was developed to calculate the measurement uncertainty. Through the propagation of errors technique, the method accounts for the uncertainty from the individual components of the leak test that are used in determining the final reported leak rate. Each step of the method is described in the following sections.

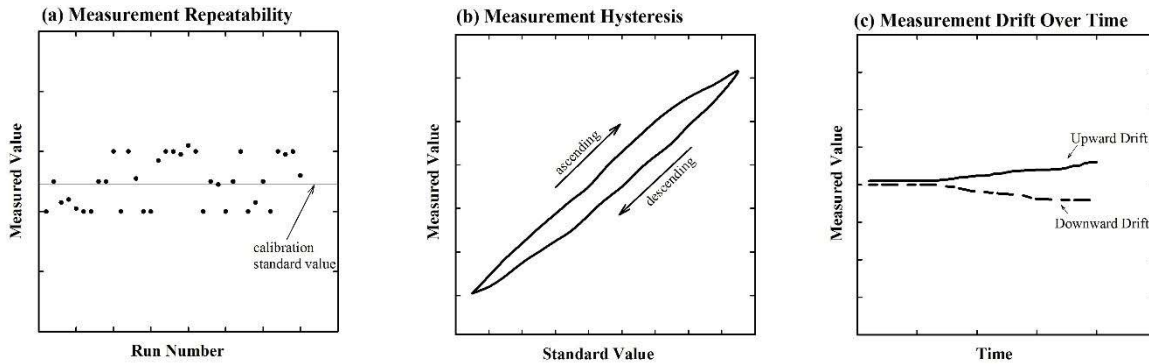


Figure 1: Graphical representations of measurement a) repeatability, b) hysteresis, and c) drift which are used along with resolution to characterize the uncertainty of a mass spectrometer leak detector.

Step 1 – Characterization of the Helium Mass Spectrometer Leak Detector

The first step is to characterize the helium MSLD with respect to the four measurement uncertainty components of resolution, repeatability, hysteresis, and drift. It is unusual to find specifications for resolution, repeatability, hysteresis, and drift for a MSLD, so these must be determined experimentally. Once these are determined for a given MSLD unit, the values are valid until the next calibration cycle or until a different measurement range is needed (see Case 1 and Case 2 in the Application Section for an example). The resolution of the measurement is directly related to the displayed value, and is a precision component of uncertainty. It represents half the range of the rightmost digit after the decimal that is not displayed. For a display of $\#.\#E-05$ scc/s the resolution is $\#.\#5E-05$ scc/s, likewise, for $\#.\#\#E-07$ scc/s the resolution is $\#.\#\#5E-07$ scc/s. Repeatability is the other precision component and is the “closeness of the agreement between the results of successive measurements”.⁹ Repeatability is determined by placing a calibration standard in the test system and recording the resulting measurement. This process is repeated

multiple times under the same conditions. The standard deviation of the group of measurements is calculated and then multiplied by the corresponding t-statistic from the Student's t-distribution for a two-sided test with a 95% confidence level. A minimum of 31 measurements is recommended. It should be noted that the response of a MSLD to leak rates over multiple decades of measurement is not necessarily linear, therefore, the calibration leak standard selected for the leak test should be sized correctly. For example, an E-08 leak should not be used as the calibration for a test with expected leak rates in the E-04 range. To determine hysteresis, the MSLD is used to measure calibration standards over a range of leak rates. Typically the range is within a single decade of measurement and is representative of the expected leak test decade of measurement. First the standards are measured in order from smallest to largest. The order is then reversed and the standards are measured largest to smallest. The maximum difference between the ascending and descending points at each calibration standard leak rate is taken as the hysteresis bias error. The final component, drift, is another bias component which provides information on how well the MSLD continuously measures a given value for a duration of time. With the calibration standard in the test system and the MSLD operating, measurements are recorded over a time period that meets or exceeds the expected test duration. Both the measured leak rate value and the time at which the measurement was recorded are documented. Similar to hysteresis, the maximum absolute difference between the calibration standard value and the resulting measured values is the drift. Examples of visual representations of these components are given in Figure 1. Both the precision ($\sigma_{\text{MSLD}\phi}$) and bias ($\sigma_{\text{MSLD}\beta}$) of the MSLD are calculated using the RSS method to sum the individual precision and bias components.

Step 2 – Characterization of the Calibration Standard

After the MSLD is characterized, the next step is to characterize the calibration standard's uncertainty. The uncertainty of the calibration standard and its temperature compensation are specified by the manufacturer or calibrating company. When temperature compensation is required, the uncertainty is determined using the propagation of errors (Equation 1). The uncertainty ($\sigma_{\text{calstd}\beta}$) is a bias error as it is an indicator of the degree to which the measured value could vary from the stated calibrated value.

Calibration standards are classified as one of two types: reservoir standards or non-reservoir standards, which are also known as closed and open standards, respectively. In reservoir type standards, the helium supply is contained within the standard, whereas in the non-reservoir standards, helium must be supplied to the standard at the specified pressure with the specified purity of gas. Therefore, in addition to the uncertainty of the calibration standard itself, the uncertainty of the pressure measurement device(s) must be accounted for and included in the total uncertainty of the calibration standard. The pressure measurement device(s) also have a precision component of repeatability that must be tracked as the calibration standard leak rate precision uncertainty ($\sigma_{\text{Qstd}\phi}$) and included in the propagation of errors for the correction factor (see Step 4).

Step 3 – Characterization of the Leak Rate System

The previous two steps can be applied to each individual leak test performed with a given test system or set-up. In this step, Step 3, the leak rate test itself is performed. Measurements are taken and recorded for each of the following test phases: pre-test calibration standard, leak rate test, and post-test calibration standard. The helium supply pressure is also taken and recorded when applicable.

Step 4 – Calculate the Test Measurement Uncertainty

Once the leak test has been completed, the data can be analyzed and the measurement uncertainty calculated. In this step, the correction factors, the reported leak rate, and all associated uncertainties are determined.

Correction Factor Calculations

In dynamic system leak testing, correction factors are used as part of the calibration of the leak test system. The calibration standard is attached to the system at the point furthest from the inlet to the MSLD. The MSLD measured leak rate is compared to the leak rate of the calibration standard, and a correction factor is used to adjust the measured leak rate to the calibration standard leak rate. A simple correction factor equation is $K = Q_{\text{std}}/Q_{\text{sm}}$ where K is the correction factor, Q_{std} is the calibration standard leak rate as stated by the manufacturer, and Q_{sm} is the MSLD output (i.e., the measured leak rate). Often the correction factor is more complex than this and is based on correction factors taken at the beginning and end of a test. For the calculations reported here, Q_{std} is an open type calibration standard with a pressure transducer. The leak rate is calculated using the input helium supply pressure (P) and the

manufacturer's equation and pressure term coefficients (Equation 3). The correction factor K is the average of the initial and final correction factors, K_i and K_f (Equation 4).

$$\text{Equation 3} \quad Q_{\text{std}} = a_2 P^2 + a_1 P + a_0$$

$$\text{Equation 4} \quad K_i = \frac{Q_{\text{std}i}}{Q_{\text{sm}i}} \quad \text{and} \quad K_f = \frac{Q_{\text{std}f}}{Q_{\text{sm}f}}$$

Correction Factor Uncertainty Calculations

The propagation of errors as stated in Equation 1 is used to calculate the bias and precision uncertainty components of Q_{std} and K_i . Equations 5 and 6 are specific for the bias contribution to uncertainty. The precision contributions have similar equations where the uncertainty terms (σ) are precision uncertainties. Recall that $\sigma_{\text{calstd}\beta}$ is a bias term without a corresponding precision term. The MSLD uncertainty components calculated in Step 1 are the uncertainty terms applied to the measured leak rate of the standard; that is $\sigma_{\text{MSLD}\beta} = \sigma_{Q_{\text{sm}\beta}}$, and $\sigma_{\text{MSLD}\phi} = \sigma_{Q_{\text{sm}\phi}}$. Equations 5 and 6 are written specifically for the pre-test calibration and the initial correction factor. The post-test calibration and the final correction factor equations are the same except the subscript i is replaced with the subscript f .

$$\text{Equation 5} \quad \sigma_{Q_{\text{std}i}\beta} = \sqrt{(2 \cdot a_2 \cdot P_i + a_1)^2 (\sigma_{PT\beta})^2 + (\sigma_{\text{calstd}\beta})^2}$$

$$\text{Equation 6} \quad \sigma_{K_i\beta} = \sqrt{\left(\frac{1}{Q_{\text{sm}i}}\right)^2 (\sigma_{Q_{\text{std}i}\beta})^2 + \left(\frac{Q_{\text{std}i}}{Q_{\text{sm}i}^2}\right)^2 (\sigma_{\text{MSLD}\beta})^2}$$

Reported Leak Rate Calculations

How the correction factor is applied to the measured leak rate of the test is specified in the leak test procedure. For the leak test described here, the measured leak rate is multiplied by the correction factor to obtain the reported leak rate, $Q_r = Q_m \cdot K$.

Reported Leak Rate Uncertainty Calculations

Equation 1 is used again to calculate the bias and precision uncertainty components of the reported leak rate. The RSS method, Equation 3, is used to combine the bias and precision errors into one total measurement uncertainty.

$$\text{Equation 7} \quad \sigma_{Q_{r\beta}} = \sqrt{(Q_m)^2 \left(\frac{\sigma_{K_i\beta} + \sigma_{K_f\beta}}{2}\right)^2 + \left(\frac{K_i + K_f}{2}\right)^2 (\sigma_{\text{MSLD}\beta})^2}$$

$$\text{Equation 8} \quad \sigma_{Q_{r\phi}} = \sqrt{\left(\frac{Q_m}{2}\right)^2 (\sigma_{K_i\phi})^2 + \left(\frac{Q_m}{2}\right)^2 (\sigma_{K_f\phi})^2 + \left(\frac{K_i + K_f}{2}\right)^2 (\sigma_{\text{MSLD}\phi})^2}$$

$$\text{Equation 9} \quad \sigma_{Q_r} = \sqrt{(\sigma_{Q_{r\beta}})^2 + (\sigma_{Q_{r\phi}})^2}$$

Step 5 – Report the Leak Rate with Associated Measurement Uncertainty

The final step in the method is the reporting and evaluation of the leak rate. The measured leak rate is reported with its associated measurement uncertainty in the format of Leak Rate \pm Total Measurement Uncertainty, $Q_r \pm \sigma_{Q_r}$. The leak rate is then evaluated for acceptance or rejection. When evaluating the leak rate, the associated uncertainty is added to the leak rate value for requirements with upper limits (i.e., the leak rate shall not exceed requirements) and subtracted from the leak rate value for requirements with lower limits (i.e., the leak rate shall be at least requirements). For example, when the requirement is such that the leak rate cannot be greater than 4.0E-08 scc/s, the reported leak rate 3.3E-08 scc/s \pm 2.7E-09 scc/s evaluates 3.6E-08 scc/s against the requirement, and the system is deemed ACCEPTABLE.

METHOD APPLICATION

Dynamic helium MSLD tests were performed on candidate seals for spacecraft hardware. The leak rate requirements represented the maximum allowable leak rate of habitable cabin air to space. Therefore, in order to properly evaluate the leak rates, the uncertainty in the reported value was required. The tests were performed using a MSLD with a sensitivity specification of 6E-10 scc/s. Two cases are presented (Table 1) to demonstrate the

difference in measurement uncertainty while using the same MSLD unit and therefore the same sensitivity. The only difference in the uncertainty components of the MSLD is the resolution term (Table 1, Row 1) because the expected test measurement range is different for the two cases. The measurement uncertainty in Case 1 is 6.6% of the reported leak rate while the measurement uncertainty for Case 2 is 13.9% of the reported leak rate. In both cases, the precision contribution is greater than the bias contribution. The results highlight that sensitivity alone is not enough to convey information about the reliability of the measured and reported leak rate.

Table 1: Helium Leak Test Results for Two Cases Using a MSLD

	Case 1	Case 2
MSLD hysteresis (β)	$\sigma_{\text{Hyst}} = 0 \text{ scc/s}$	$\sigma_{\text{Hyst}} = 0 \text{ scc/s}$
MSLD drift (β)	$\sigma_{\text{Drift}} = 0 \text{ scc/s}$	$\sigma_{\text{Drift}} = 0 \text{ scc/s}$
MSLD resolution (ϕ)	$\sigma_{\text{Resolu}} = 5.00\text{E-}05 \text{ scc/s}$	$\sigma_{\text{Resolu}} = 5.00\text{E-}07 \text{ scc/s}$
MSLD repeatability (ϕ)	$\sigma_{\text{Repeat}} = 9.42\text{E-}05 \text{ scc/s}$	$\sigma_{\text{Repeat}} = 9.42\text{E-}05 \text{ scc/s}$
Overall Bias	$\sigma_{\beta} = 7.67\text{E-}05 \text{ scc/s}$	$\sigma_{\beta} = 3.99\text{E-}06 \text{ scc/s}$
Overall Precision	$\sigma_{\phi} = 1.08\text{E-}04 \text{ scc/s}$	$\sigma_{\phi} = 1.41\text{E-}05 \text{ scc/s}$
Total Measurement Uncertainty	$\sigma_Q = 1.33\text{E-}04 \text{ scc/s}$	$\sigma_Q = 1.46\text{E-}05 \text{ scc/s}$
Reported Leak Rate Q	$Q_r = 2.01\text{E-}03 \text{ scc/s} \pm 1.33\text{E-}04 \text{ scc/s}$	$Q_r = 1.05\text{E-}04 \text{ scc/s} \pm 1.46\text{E-}05 \text{ scc/s}$

SUMMARY

To provide an indication of the reliability of the leak rate from a MSLD, a method for determining the total measurement uncertainty was developed for use in these tests. The method uses the concept of propagation of errors to account for the measurement uncertainty associated with the leak detector unit itself, the calibration standard, and the correction factor. The leak detector unit is characterized with respect to its resolution, repeatability, drift, and hysteresis instead of the sensitivity. In application, it was found that the precision component contributed more to the total uncertainty than the bias component, but the bias component was not insignificant.

REFERENCES

1. American Vacuum Society, "Calibration of Leak Detectors of the Mass Spectrometer Type", *J. Vacuum Science and Technology*, Vol. 10(4), Jul/Aug 1973.
2. American Society for Testing and Materials, "Standard Terminology for Nondestructive Examinations", ASTM E1316-14, West Conshohocken, PA, 2014.
3. Jackson, Charles N., Jr., and Charles N. Sherlock, *Nondestructive Testing Handbook*, third edition: Volume 1 *Leak Testing*, Columbus, OH, American Society for Nondestructive Testing, 1998.
4. American Society for Testing and Materials, "Standard Practice for Leaks Using the Mass Spectrometer Leak Detector in the Inside-Out Testing Mode", ASTM E493-E493M-11, West Conshohocken, PA, 2011.
5. American Society for Testing and Materials, "Standard Practice for Leaks Using the Mass Spectrometer Leak Detector or Residual Gas Analyzer in the Tracer Probe Mode", ASTM E498/E498M-11, West Conshohocken, PA, 2011.
6. American Society for Testing and Materials, "Standard Practice for Leaks Using the Mass Spectrometer Leak Detector in the Detector Probe Mode", ASTM E499/E499M-11, West Conshohocken, PA, 2011.
7. Varian Vacuum Products, *Introduction to Helium Mass Spectrometer Leak Detection*, second edition, 1995.
8. Pryor, Jeff M., and William C. Walker, "Pressure Change Measurement Leak Testing Errors", *Materials Evaluation*, Vol. 72, May 2014.
9. American National Standard for Calibration, *U.S. Guide to the Expression of Uncertainty in Measurement*, ANSI/NCSL Z540-2-997 (R2007).
10. Bevington, Philip R., and D. Keith Robinson, *Data Reduction and Error Analysis for the Physical Sciences*, third edition, McGraw-Hill, New York, 2003.

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